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Facile synthesis of flake-like nickel disulfide nanocrystallites

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ABSTRACT

Pure cubic pyrite flake-like nickel disulfide nanocrystallites have been successfully synthesized by thermal decomposition approach under open-air conditions at 280 °C using bis(thiourea) nickel (II) chloride crystals as precursors. The crystallinity and morphology of the products were found to be dependent on the reaction temperature. Raman spectroscopy study indicates that peak of flake-like nickel disulfide located at 478.7 cm^{-1} should correspond to a stretching vibration with an A_g mode. Magnetization measurement indicates that as-prepared product at 280 °C under open-air condition displays anomalous magnetic property at room temperature and shows weak ferromagnetic. The present thermal approach in a complex system may allow synthesizing other metal chalcogenides by decomposing under open-air conditions.

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1. Introduction

Among the family of nickel sulfides, nickel disulfide, a pyrite structure [1], famous as a Mott–Hubbard insulator with a band gap of about 0.3 eV [2–4] and a metal–insulator transitional body in $NiS_{2-x}Se_x$ [5–7], has attracted considerable interest due to its electrical, optical, semiconducting and magnetic properties [8–14]. These properties have aroused intensive investigations for new applications including use in Dye-Sensitized Solar Cells [15], catalysts for selective H_2S oxidation [16,17], catalysts for hydrodesulfurization [18] and for hydrogen evolution reaction [19], cathode materials for lithium-ion batteries [20] and magnetic devices [21].

Recently, many methods have been introduced to synthesize nickel disulfide nanocrystallines. For instance, NiS_2 nanocrystallites can be prepared by a rapid low-temperature solid-state method [22,23], solvothermal or hydrothermal method [24–30], microwave assisted hydrothermal technique [31], cyclic microwave radiation [32], ultrasonic spray pyrolysis technique [33], CBD technique [34], a directional infiltration self-assembly route [35], irradiation and chemical nanofabrication methods [36–38].

Here, we report a facile method for preparing cubic pyrite flake-like NiS_2 nanocrystallites through decomposing bis(thiourea) nickel (II) chloride crystals under open-air conditions. It is to be noted that the flake-like NiS_2 nanocrystallites are synthesized without any protect gas and in a relative low temperature compared to those reported by Kumar et al. [39]. And it is the first time to take bis(thiourea) nickel (II) chloride crystals as the precursor to synthesis the nickel disulfide. The present facile approach in a complex system may allow synthesizing other metal chalcogenides. Information of the samples was investigated using X-ray diffraction (XRD), Field emission scanning electron microscopy (FESEM), Transmission electron microscopy (TEM) images, Raman spectrum and Fourier transform infrared (FTIR) spectrum; meanwhile

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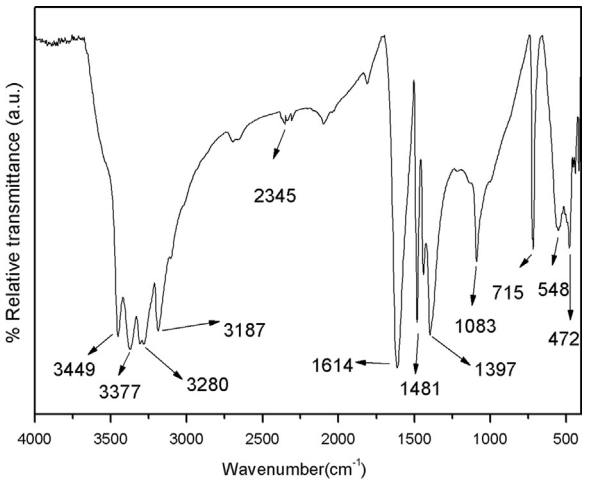

Fig. 1. FTIR spectrum of bis(thiourea) nickel (II) chloride crystals.

magnetic property of the pyrite NiS_2 was studied using a vibrating sample magnetometer (VSM).

2. Experimental

All the analytical chemicals were purchased from Shanghai Chemical Reagents Company and used without further purification.

2.1. Synthesis of bis(thiourea) nickel (II) chloride crystals

Bis(thiourea) nickel (II) chloride crystals was synthesized as the following reaction using thiourea and nickel chloride mixed in stoichiometric ratio of 1:2 according to an earlier reported method [40]:

$$NiCl_2 \cdot 6H_2O + 2[CS(NH_2)_2] \rightarrow Ni[CS(NH_2)_2]_2Cl_2 \quad (9)$$

In a typical synthesis, 1.9 g thiourea and 2.9 g nickel chloride salts were dissolved in the solvent of 20 ml distilled water, and then the solution was allowed to evaporate at room temperature about 30 °C for 2 days. Good quality crystals were obtained.

2.2. Synthesis of flake-like NiS_2 nanocrystallites

In a typical experimental procedure for fabrication of flake-like NiS_2 nanocrystallites, the as-prepared bis (thiourea) nickel (II) chloride crystals were transferred into a 30 ml ceramic pot. The pot was unsealed and maintained at 280 °C for 20 h under open-air conditions. After cooling to room temperature naturally, the dark products were obtained, washed with distilled water and absolute ethanol for several times, and dried in air atmosphere at 50 °C for 10 h. For comparison, precursors with different reaction temperatures were carried on while keeping other reaction conditions unchanged.

2.3. Characterization and property of samples

Bis(thiourea) nickel (II) chloride crystals were characterized by a KYKY2800B scanning electron microscope (SEM) equipped with Energy-dispersive X-ray (EDX) and a Perkin-Elmer Spectrum One FTIR Spectrometer using a KBr-pellet technique at a resolution of 1 cm^{-1} in the range of 400–4000 cm^{-1}. And for the flake-like NiS_2 nanocrystallites, X-ray powder diffraction (XRD) patterns were recorded on a XD-2 X-ray diffractometer with Cu Kα radiation ($\lambda = 1.54187$ Å) in the 2θ range of 10–70°. SEM images were taken with an Inspect F50 field emission scanning electron microscope (FESEM), Transmission electron microscopy (TEM) images were obtained on a Tecnai G2 20 transmission electron microscope with accelerating voltage of 200 kV. The Fourier-transform infrared (FTIR) spectra were measured with Perkin-Elmer Spectrum One FTIR Spectrometer using the KBr-pellet technique in the range 400–4000 cm^{-1}. The Raman spectrum was measured with a Renishaw Invia laser Raman spectrometer. The magnetic measurement was carried out in a vibrating sample magnetometer (VSM) (MPMS-XL-7) at room temperature.

3. Results and discussion

The FTIR spectrum of bis(thiourea) nickel (II) chloride crystals is recorded at a resolution of 1 cm^{-1} in the frequency region of 4000–400 cm^{-1} as shown in Fig. 1. The broad envelope positions in between 3150 and 3500 cm^{-1} corresponds to the symmetric and asymmetric stretching modes of $-NH_2$ group. The absorption band observed at 1614 cm^{-1} is assigned to NH_2 bending mode of vibrations. The CN stretching frequencies of thiourea (1081 and 1472 cm^{-1}) are shifted to 1083 and 1481 cm^{-1} for the complex. The slight shift in the frequency can be attributed to the carbon to nitrogen bond on complex formation. The symmetric and asymmetric stretching frequencies of C=S (1417 and 740 cm^{-1}) for thiourea are shifted to lower frequencies (1397 and 715 cm^{-1}) for the complex. Lower shift of C=S stretching frequency (740 to 715 cm^{-1}) confirms the formation of metal–sulfur coordination bond [41]. Therefore, bands at 548 and 472 cm^{-1} can be attributed to the Ni–S stretching vibration modes of the bis(thiourea) nickel (II) chloride crystals.

Fig. 2a–c shows the SEM images of as-prepared bis (thiourea) nickel (II) chloride crystals using thiourea and nickel chloride mixed in the stoichiometric ratio of 1:2 by a slow evaporation solution growth technique at room temperature. It can be seen that the products mainly consist of tetrakis prismatic like crystal structures with different diameters range 0.1 mm to 1.0 mm as shown in Fig. 1a–b. Energy dispersive X-ray (EDX) analysis indicates that both nickel and sulfur elements are detected with a molar ratio close to 1:2 (Fig. 2d), which is consistent with the stoichiometric composition. On the basis of the above FTIR and EDX analyses, it suggested the formation of bis (thiourea) nickel (II) chloride crystals samples.

Fig. 3a–c shows XRD patterns of the NiS_2 nanocrystallites obtained at different temperatures for 20 h under open-air conditions. All peaks correspond to NiS_2 phase

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